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Indian Standard
SPECIFICATION FOR
AMYL SALICYLATE
(*First Revision*)

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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Indian Standard

SPECIFICATION FOR AMYL SALICYLATE

(First Revision)

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Indian Standard

SPECIFICATION FOR AMYL SALICYLATE

(First Revision)

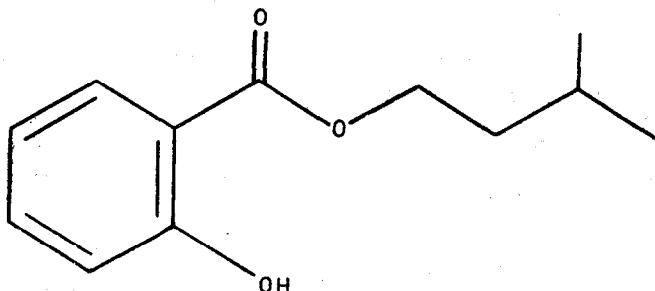
0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 16 August 1984, after the draft finalized by the Natural and Synthetic Perfumery Materials Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 This standard was first published in 1966. The Sectional Committee felt that the standard should be revised with a view to bring it in line with trade practices in perfumery technology and also align with quality level of the material currently manufactured and sold in the country.

0.3 In this revision, the gas chromatographic method which is being progressively used in the country has been included and the wet method of analysis for determination of ester content has been deleted. Requirements for relative density and refractive index have also been modified.

0.4 Amyl salicylate ($C_{12}H_{16}O_3$) is not found in nature. Commercial amyl salicylate chemically is mainly *iso*-amyl salicylate. It is also known as *iso*-amyl *o*-hydroxy-benzoate. It finds extensive application in compounding of floral odours, specially for use in soaps. It has a very lasting, treble type odour. It is used in a wide variety of compositions because of its low price and lasting quality. It is represented by the following structural formula.



AMYL SALICYLATE
(Molecular Mass 208.25)

0.5 In the preparation of this standard, considerable assistance has been derived from the following publications:

The Givaudan Index, 1978, published by Givaudan — Delawanna Inc., New York.

EOA No. 27-1970 Standard for amyl salicylate (*revised*), published by Essential Oil Association of USA, New York.

0.6 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for amyl salicylate.

2. TERMINOLOGY

2.1 For the purpose of this standard, definitions given in IS : 6597-1972† shall apply.

3. REQUIREMENTS

3.1 Description

3.1.1 The material shall be obtained by esterification of *iso*-amyl alcohol with salicylic acid.

3.1.2 The material shall be a colourless liquid free from sediment, suspended matter and adulterants.

3.1.3 The material shall be examined for its colour, clarity, suspended matter and sediments when examined by the method prescribed in IS : 326 (Part 2)-1980‡.

*Rules for rounding off numerical values (*revised*).

†Glossary of terms relating to natural and synthetic perfumery materials.

‡Methods of sampling and test for natural and synthetic perfumery materials: Part 2 Preliminary examination of perfumery materials and samples (*second revision*).

3.1.4 The material shall also be tested olfactorily and especially for bye notes as prescribed under 4 and 5 of IS : 2284-1963*.

3.2 Solubility — The material shall be clearly soluble in 2 volumes of ethanol (90 percent by volume), when tested as prescribed under 8 of IS : 326-1968†.

3.3 The material shall also comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR AMYL SALICYLATE

(Clauses 3.3, 5.3.1 and 6.1)

Sl. No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST, REF TO	
			Indian Standard	Appendix
(1)	(2)	(3)	(4)	(5)
i)	Odour	Agreeable, aromatic odour recalling that of orchids	IS : 2284-1963*	—
ii)	†Relative density at 27/27°C	1.046 8 to 1.051 8	IS : 326 (Part 3)-1980†	—
iii)	§Refractive index at 27°C	1.503 3 to 1.505 3	7 of IS : 326-1968	—
iv)	Acid value, Max	0.5	IS : 326 (Part 7)-1980‡	—
v)	Amyl salicylate, per-cent by mass, Min	99	—	A

*Method for olfactory assessment of natural and synthetic perfumery materials.

†The correction factor for relative density for each degree celcius change in temperature is 0.000 61.

‡Methods of sampling and test for natural and synthetic perfumery materials:

Part 3 Relative density (*second revision*).

Part 7 Determination of acid value (*second revision*).

§The correction factor for refractive index for each degree celsius change in temperature is 0.000 42.

||Methods of sampling and test for natural and synthetic perfumery materials (*first revision*).

4. PACKING AND MARKING

4.1 Packing — The material shall be packed in glass-bottles, tins or any other suitable containers as agreed to between the purchaser and the supplier.

NOTE — Tin, iron and galvanized containers shall not be used for packing of amyl salicylate.

*Method for olfactory assessment of natural and synthetic perfumery materials.

†Methods of sampling and test for natural and synthetic perfumery materials (*first revision*).

4.2 Marking — Each container so filled shall bear legibly and indelibly the following information:

- a) Name of the material;
- b) Name of the manufacturer and his recognized trade-mark, if any;
- c) Batch number and date of manufacture; and
- d) Net and gross mass.

4.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act, and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

5. SAMPLING

5.1 Representative samples of the material shall be drawn as prescribed in IS : 326 (Part 1)-1984*.

5.2 Number of Tests

5.2.1 Amyl salicylate content shall be tested on each of the individual samples.

5.2.2 Tests for determination of all the remaining characteristics shall be conducted on the composite sample.

5.3 Criteria for Conformity — The lot shall be declared as conforming to the requirements of the specification if **5.3.1** and **5.3.2** are satisfied.

5.3.1 For ester content, the mean (\bar{X}) and range (R) of test results shall be calculated:

$$\text{Mean (} \bar{X} \text{)} = \frac{\text{Sum of the test results}}{\text{Number of the test results}}$$

$$\text{Range (} R \text{)} = \frac{\text{Difference in the maximum and minimum of the test results}}{\text{Number of the test results}}$$

*Methods of sampling and test for natural and synthetic perfumery materials:
Part I Sampling (*second revision*).

The lot shall be deemed to have satisfied the requirement for this characteristic if the value of the expression ' $X - 0.6 R$ ' is greater than or equal to the minimum limit for amyl salicylate content given in Table 1.

5.3.2 All the test results on the composite sample meet the relevant specification requirements.

6. TEST METHODS

6.1 Tests shall be carried out as prescribed under **3.1**, **3.2** and the appropriate references specified in col 4 and 5 of Table 1.

6.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070 - 1977*) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A P P E N D I X A

[Table 1, Item (v)]

GAS CHROMATOGRAPHIC ANALYSIS FOR DETERMINATION OF AMYL SALICYLATE CONTENT

A-0. GENERAL

A-0.1 The chromatographic conditions given here are for guidance only.

A-0.2 Outline of the Method — A sample of the material is dissolved in a suitable solvent (for example, cyclohexane and diethyl ether) and is injected into the gas chromatograph where it is carried by the carrier gas from one end of the column to the other. During its movement the constituents of the sample undergo distribution at different rates and ultimately get separated from one another. The separated constituents emerge from the end of the column one after another and are detected by suitable means, response of which is related to the amount of a specific component leaving the column.

A-1. APPARATUS

A-1.1 Any gas chromatograph capable of being operated under conditions suitable for resolving the individual constituents into distinct peaks may be used. The typical chromatogram for amyl salicylate using a chromatograph with the following chromatographic conditions is shown in Fig. 1.

*Specification for water for general laboratory use (*second revision*).

<i>Sample Column</i>		Amyl salicylate
a) Material	:	Copper
b) Length	:	5.49 m
c) O.D.	:	0.635 cm
d) I.D.	:	0.476 cm
e) Stationary phase	:	FFAP*, 10 percent by mass
f) Solid support	:	Chromosorb WAW 60-80 mesh

Carrier Gas Conditions : Hydrogen

- | | | |
|---|---|------------------------|
| a) Column temperature <i>iso</i> -thermal | : | 200°C |
| b) Injection port temperature | : | 200°C |
| c) Carrier gas flow rate | : | 40 ml/min |
| d) Inlet pressure | : | 3.5 kg/cm ² |

Detector

- | | | |
|----------------|---|----------------------|
| a) Type | : | Thermal conductivity |
| b) Temperature | : | 285°C |

Recorder

- | | | |
|----------------|---|--------------|
| a) Span | : | 1 mV |
| b) Chart speed | : | 0.635 cm/min |

Attenuation 4

NOTE — This analysis may also be accomplished with columns containing: Carbowax-20M, DE.G.S (Diethylene Glycol Succinate).

A-2. PROCEDURE

A-2.1 Conduct the flow of the carrier gas and inject the sample (dissolved in the suitable solvent) at injection port where it is vaporized and well mixed with the carrier gas. This is led into the chromatographic column wherein vaporized constituents of the sample are separated out by virtue of their differing interaction with the stationary phase. As the different constituents pass through the detector, they give signals corresponding to the amount of particular constituents leaving the column. The detector signals; on transmission to the recorder, plots the chart. From the specific area under various peaks corresponding to specific constituents, the quantities of different constituents are determined.

NOTE — For the separation to be efficient, it is necessary that the column is maintained at the temperature suggested throughout the time required for the resolution of the constituents.

*Free fatty acid phase (FFAP) is carbowax 20M treated with nitrophthalic acid.

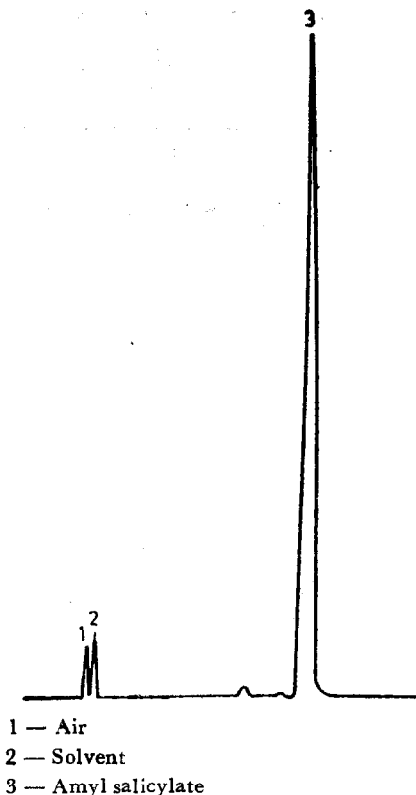


FIG. 1 TYPICAL CHROMATOGRAM OF AMYL SALICYLATE

A-3. CALCULATION

A-3.1 Area Measurement (see Note 1) — Since normal peaks approximate a triangle the area is measured by multiplying the peak height times the width of half height. The normal peak base is not taken since large deviations may be observed due to tailing or adsorption. This technique is rapid, simple and fairly accurate when peaks are symmetrical and of reasonable width.

A-3.2 Area Normalization (see Note 2) — By normalizing, it is meant, calculating the percentage composition by measuring the area of each and dividing the individual areas by total area, for example,

$$\text{Percentage of } A = \frac{\text{Area of } A}{\text{Total area}} \times 100$$

NOTE 1 — Other methods of area measurements, namely, Triangulation, Disc Integrator and Electronic Digital Integrator if fixed with GLC machine would be of great advantage.

NOTE 2 — Internal standardization can be used if pure appropriate internal standard is available. This method is known as relative or indirect calibration.

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INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

QUANTITY	UNIT	SYMBOL
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

QUANTITY	UNIT	SYMBOL
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

QUANTITY	UNIT	SYMBOL	DEFINITION
Force	newton	N	1 N = 1 kg.m/s ²
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m ²
Frequency	hertz	Hz	1 Hz = 1 c/s (s ⁻¹)
Electric conductance	siemens	S	1 S = 1 A/V
Electromotive force	volt	V	1 V = 1 W/A
Pressure, stress	pascal	Pa	1 Pa = 1 N/m ²



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